

(19)



Europäisches Patentamt  
European Patent Office  
Office européen des brevets

(11) Publication number:

9198  
0 117 070  
A2

(12)

## EUROPEAN PATENT APPLICATION

(21) Application number: 84300373.2

(51) Int. Cl.<sup>3</sup>: A 61 K 7/32

(22) Date of filing: 23.01.84

(30) Priority: 26.01.83 US 460973

(43) Date of publication of application:  
29.08.84 Bulletin 84/35(84) Designated Contracting States:  
AT BE CH DE FR GB IT LI NL(71) Applicant: THE PROCTER & GAMBLE COMPANY  
301 East Sixth Street  
Cincinnati Ohio 45201(US)(72) Inventor: May, William George  
4869 Beechwood Road  
Cincinnati Ohio 45244(US)(74) Representative: Brooks, Maxim Courtney et al,  
Procter & Gamble (NTC) Limited Whitley Road  
Longbenton  
Newcastle-upon-Tyne NE12 9TS(GB)

(54) Cosmetic sticks.

(57) Solid stick compositions comprising a long-chain fatty alcohol having from about 8 to about 18 carbon atoms in the chain, volatile silicone and C<sub>20</sub> or longer chain alcohols at levels of from about 1% to about 3% of the total long-chain fatty alcohol present in the compositions. These solid sticks have improved strength and hardness without undue brittleness.

EP 0 117 070 A2

BEST AVAILABLE COPY

COSMETIC STICKS

William G. May

The present invention relates to solid stick compositions, particularly solid antiperspirant sticks, having satisfactory strength and hardness while exhibiting aesthetically-pleasing attributes.

Many solid stick compositions are available in the art. Typically, these solid sticks are available in two basic forms. One such form is composed of soap/alcohol gel sticks, which may have stability problems. For instance, the combination of soap/alcohol gels with conventional astringent antiperspirant salts, such as aluminium chlorohydrate, can result in unstable gel structures rendering such sticks less aesthetically-pleasing to consumers.

In order to alleviate some of these problems, waxy materials, some in combination with volatile fluids, have been used in the development of solid stick compositions. US-A-4,126,679, November 21, 1978 to Davy and Drolet; US-A-4,229,432, October 21, 1980 to Geria; US-A-4,265,878, May 5, 1981 to Keil; US-A-4,280,994, July 28, 1981 to Turney disclose such compositions. The most commonly used waxy material in sticks of this type include long-chain fatty alcohols. Some use such alcohols in combination with volatile silicones. Although these fatty alcohol/volatile silicone solid sticks avoid some of the soap gel problems, they may vary unacceptably in structural hardness and strength. Therefore, formulations are sought which provide strong fatty alcohol/volatile silicone-based sticks without causing those sticks to become too brittle and cosmetically unacceptable.

Surprisingly, the addition of fatty alcohols, C<sub>20</sub> and longer, produces such a product. That is, not only is the strength of such a stick increased, but that strength enhancement does not result in overly hard, brittle product.

5 It is an object, therefore, of the present invention to provide solid stick compositions containing long-chain higher fatty alcohols and a volatile silicone wherein such compositions contain sufficient C<sub>20</sub> alcohol and longer chain alcohols to result in a stable and strong final product.

It is a further object of the present invention to not only provide strong final products but to provide aesthetically-pleasing products as well.

10 Another object of this invention is to provide solid stick compositions as described above which additionally contain astringent antiperspirant salt materials.

These and other objectives will become more apparent from the following disclosure. All percentages and ratios herein are by weight unless otherwise designated.

15 SUMMARY OF THE INVENTION

The solid stick compositions of the present invention are comprised of from about 5% to about 20% of a long-chain fatty alcohol and from about 35% to about 55% volatile silicone, wherein said compositions also contain an alcohol selected from the group  
20 consisting of C<sub>20</sub> alcohol, alcohols whose chains are longer than C<sub>20</sub> and mixtures thereof at levels of from about 1% to about 3% of the total long chain fatty alcohols present in said compositions.

DETAILED DESCRIPTION OF THE INVENTION

25 The present solid stick compositions contain certain essential and may in addition contain other nonessential, optional components. Each component is discussed in detail below.

LONG-CHAIN HIGHER FATTY ALCOHOL

30 The higher fatty alcohols of the present invention are those having melting points of from about 100°F. to about 150°F. These include fatty alcohols containing from about 8 to about 18 carbon atoms in their chain, preferably from about 12 to about 18 carbon atoms. Examples suitable for use in the invention at hand include cetyl alcohol, stearyl alcohol, myristyl alcohol, lauryl alcohol, and mixtures thereof.

35 From about 5% to about 20% higher fatty alcohol is present

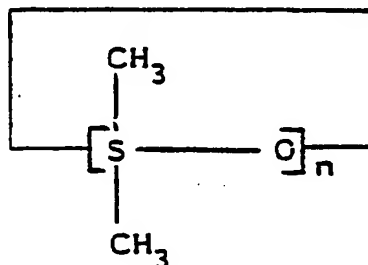
in the solid stick compositions, preferably from about 10% to about 15%.

As indicated above, the compositions described herein also contain from about 1% to about 3%, based on the total weight of the long chain fatty alcohols present (the  $C_{12}$  -  $C_{18}$  alcohols as well as the longer chain alcohols), of alcohols having chain lengths of  $C_{20}$  or longer. A preferred alcohol of this type is commercially available  $C_{20}$  alcohol which is a mixture of higher fatty alcohols wherein about 50% is  $C_{20}$ , about 30% is  $C_{22}$ , with the remainder primarily composed of alcohol chains longer than  $C_{22}$ . The alcohols useful in the present invention may go up to  $C_{26}$  but it is preferred to have the majority in the  $C_{20}$  -  $C_{22}$  range. The level of  $C_{20}$  and longer chain alcohols found in the commercially available shorter long-chain fatty alcohols (e.g. stearyl alcohol) generally does not exceed about 0.8%. Only by increasing the level of these alcohols to from about 1% to about 3%, preferably from about 1% to 2%, of the total higher fatty alcohol content will a stronger product result.

#### VOLATILE SILICONE

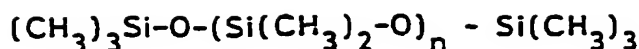
The volatile silicones useful in the solid stick compositions of the present invention may be either cyclic or linear with the polydimethylsiloxanes containing from about 3 to about 9, preferably from about 4 to about 5, silicon atoms, being preferred.

The following formula illustrates the cyclic volatile polydimethylsiloxanes useful in the solid stick compositions disclosed herein:



wherein  $n = 3$  to 7.

The linear polydimethylsiloxanes contain from about 3 to about 9 silicon atoms per molecule and have the following general formula:



wherein  $n = 1$  to  $7$ .

5           Examples of silicones of the above type include those offered by Dow Corning Corporation, Dow Corning 344, 345 and 200 fluids, Union Carbide, Silicone 7207 and Silicone 7158, and Stauffer Chemical, SWS-03314.

10           The linear volatile materials generally have viscosities of less than about 5 centistokes at  $25^\circ\text{C}$ . while the cyclic materials have viscosities less than about 10 centistokes. "Volatile" means that the material has a measurable vapor pressure. A description of volatile silicones is found in Todd and Byers, "Volatile Silicone Fluids for Cosmetics", Cosmetics and Toiletries, Vol. 91, January, 15 1976, pp. 27 - 32, incorporated herein by reference.

          The amount of volatile silicone used in the present compositions is from about 35 to about 55, preferably from about 40 to about 50%.

#### OPTIONAL COMPONENTS

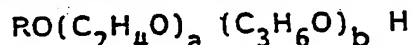
20           The solid stick compositions of the present invention may contain a variety of optional, nonessential components. These components serve a variety of functions such as improving the stability, cosmetics and/or aesthetics of the present compositions.

#### WAX

25           A preferred optional component includes a wax such as castor wax, fatty acids, silicone waxes and glycerol monostearate, and mixtures thereof at levels of from about 1% to about 10%, preferably from about 3% to about 10%. If present, the wax is believed to enhance structural stability at higher temperatures.

EMOLLIENT

The emollient component of the present invention is useful in providing an aesthetically-pleasing product. One type of emollient suitable for use in the present solid stick compositions is an ethylene oxide and/or propylene oxide condensation product having the following formula:



wherein R is either hydrogen or a hydrocarbon chain having from about 2 to 20 carbon atoms, preferably from about 4 to about 18, a and b are each from about 0 to about 35, and a+b is from about 5 to about 35.

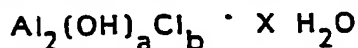
Examples of such emollients include Fluid AP<sup>®</sup> (a condensate of about 14 moles of propylene oxide with about 1 mole of butyl alcohol sold by Union Carbide); a polypropylene glycol having molecular weight of about 1200; a polyethylene glycol having molecular weight of about 420; a condensate of 20 moles ethylene oxide and 5 moles propylene oxide with one mole of cetyl alcohol, and mixtures thereof.

Other emollients suitable for use in the present solid stick compositions include fatty acid and fatty alcohol esters and water insoluble ethers such as those disclosed in U.S. Patent 4,202,879, May 13, 1980 to Shelton, incorporated herein by reference and mixtures thereof.

If present, the emollient is used at levels of from about 2% to about 10%, preferably from about 3% to about 7%.

ASTRINGENT ANTIPERSPIRANT

Any aluminum astringent antiperspirant salt or aluminum and/or zirconium complex can be employed herein when the present sticks are intended for use as an antiperspirant. Useful are such salts as aluminum halides, aluminum hydroxyhalides, zirconyl oxyhalides, zirconyl hydroxyhalides, and mixtures thereof. The aluminum salts of this type include aluminum chloride and aluminum hydroxyhalide having the following formula:



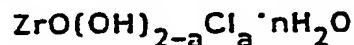
a is from about 2 to about 5;  
a+b = 6 and a and b need not be integers; and x is from about 1 to about 6. The manner of preparing such salts is disclosed in

U.S. Patent 3,887,692, June 3, 1975 to Gilman, incorporated herein by reference.

Zirconium salts are also useful in the present invention. Such salts include zirconium oxychloride, zirconium hydroxychloride, and zirconium salts of the formula,  
5  $ZrO(OH)_{2-a}Cl_a \cdot nH_2O$ , wherein a is from about 1.5 to about 1.87 and n is from about 1 to about 7. These types of zirconium salts are discussed in Belgium Patent 825,146, August 4, 1975 to The Procter & Gamble Company, incorporated herein by reference.

10 Complexes of the previously-discussed astringent salts are disclosed in U.S. Patent 3,679,068, February 12, 1974 to Luedders, et al, incorporated herein by reference. The Leudders et al patent discloses complexes of aluminum, zirconium and glycine commonly known as ZAG complexes.

15 The preferred aluminum salt for use in such ZAG complexes is aluminum chlorhydroxide with the preferred zirconium compounds being zirconyl hydroxychloride and other zirconyl hydroxyhalides of the following formula:



20 wherein a is from about 1.5 to about 1.87 and n is from about 1 to about 7. U.S. Patent 4,120,948, October 17, 1978 to Shelton discusses these preferred ZAG compounds and is incorporated herein by reference.

25 Other antiperspirant complexes are also suitable for use in the present compositions. For example, U.S. Patent 3,903,258, September 2, 1975 to Siegel, discloses a zirconyl chloride/aluminum hydroxide and aluminum chlorhydroxide complex. U.S. Patent 3,979,510, September 6, 1976 to Rubino discloses complexes composed of particular aluminum, zirconium and aluminum buffer  
30 compounds. Additionally, U.S. Patent 3,981,986, September 21, 1976 to Rubino, discloses the aluminum and zirconium complexes with an organic buffer, and U.S. Patent 3,970,748, July 20, 1976 to Mecca, discloses an aluminum chlorhydroxy glycinate complex. All of these patents are incorporated herein by reference.

Best Available Copy

The levels of these antiperspirant materials range from about 10% to about 70%, preferably from about 15% to about 50%, most preferably from about 15% to about 25% on a pure antiperspirant salt basis. In the case of ZAG type materials glycine is excluded when antiperspirant salt level is determined.

#### OTHERS

Another optional component of the present invention is inert filler material. This filler material enhances the aesthetic characteristics of the present solid sticks and may serve to stabilize the structure of such sticks.

Among the filler materials suitable for use in the present invention are talc, colloidal silica such as CAB-O-Sil (Cabot Corp.), clays such as bentonite, and mixtures thereof. The inert filler material comprises from about 0.5% to about 10% of the present solid stick compositions.

Conventional deodorant materials also may be included in the present invention. Suitable deodorants include bacteriostatic quaternary ammonium compounds such as cetyl-trimethylammonium bromide, cetyl pyridinium chloride, benzethonium chloride, diisobutyl phenoxy ethoxy ethyl dimethyl benzyl ammonium chloride, sodium N-lauryl sarcosine, sodium N-palmethyl sarcosine, lauroyl sarcosine, N-myristoyl glycine, potassium N-lauryl sarcosine, stearyl, trimethyl ammonium chloride and mixtures thereof. Another suitable deodorant material is 2,4,4'-trichloro-2'-hydroxydiphenyl ether. If present, these deodorants comprise from about 0.1% to about 1.0% of the composition.

Other optional components include perfumes, pigments, dyes, coloring agents, and the like, at levels individually of from about 0.1% to about 2.0%.

#### METHOD OF MANUFACTURE

The solid stick compositions of the present invention are made in accordance with well-established methods known to those knowledgeable in the art.



INDUSTRIAL APPLICATION

The present solid stick compositions are useful as vehicles for a wide variety of cosmetic materials. For instance, a preferred use of these solid stick compositions is as antiperspirant sticks. However, these sticks can be used as deodorant sticks as well as other cosmetic vehicles.

The following example is presented as an illustration of the present invention and is not limitative thereof.

EXAMPLE I

An antiperspirant stick was made utilizing the following components:

<u>Component</u>	<u>Weight %</u>
Cyclomethicone	43.95
Fluid AP	4.99
Stearyl Alcohol	11.49
Castor Wax	4.99
Talc	6.99
Zirconium/Aluminum/Glycine complex	26.67
Fragrance Masking Agent	0.80
C <sub>20</sub> OH*	0.12

\*This is the commercial C<sub>20</sub> alcohol mixture described previously.

Best Available Copy

EXAMPLE II

5 An antiperspirant stick similar to that described in Example I was prepared but the C<sub>20</sub> and longer chain alcohol level was only 0.034%. This stick therefore did not have the required amount of such long chain alcohols.

EXAMPLE III

10 The sticks of Examples I and II were compared for softness and break strength. The softness measurements were made using a penetrometer while the break strength measurements were made using a Chatillon break strength tester. The results of these tests are shown below.

15		<u>Penetration</u>	<u>Break Strength</u>
		(Tenths of a mm)*	(Pounds Force)**
20	Example I Product	78.1	11.3
	Example II Product	96.1	7.1

25 \* The penetration values are determined by using ASTM Method D-5. The measurements are made at least 10 mm from the edge of a stick.

30 \*\* The breakstrength is determined using a Hunter Force Gauge, Model L-10, 0 - 30 lb. range. The gauge is attached to a slide which allows the gauge to contact the test stick through a breaker bar at a speed of three inches per minute. The value recorded is the force gauge reading when the stick breaks.

35 It is seen that the product of the present invention has a greater break strength while also having a lower penetration value.

WHAT IS CLAIMED IS:

CLAIMS

1. A solid stick composition possessing improved strength without undue brittleness characterized by:

(a) from 5% to 20% of a long-chain fatty alcohol having from 8 to 18 carbon atoms in its chain; and

(b) from 35% to 55% volatile silicone;

wherein said composition additionally contains an alcohol selected from  $C_{20}$  alcohol, alcohols whose chains are longer than  $C_{20}$  and mixtures thereof at a level of from 1% to 3% of the total fatty alcohol level.

2. A solid stick composition according to Claim 1 characterized in that it additionally contains from 1% to 10% of a wax selected from castor wax, fatty acids, silicone waxes, glycerol monostearate, and mixtures thereof.

3. A solid stick composition according to Claim 1 or 2 characterized in that said long-chain fatty alcohol is present at a level of from 10% to 15% and is selected from cetyl alcohol, stearyl alcohol, myristyl alcohol, lauryl alcohol, and mixtures thereof.

4. A solid stick composition according to any of Claims 1 to 3 characterized in that said volatile silicone is selected from cyclic and linear polydimethylsiloxanes having from 3 to 9 silicon atoms in their chain.

5. A solid stick composition according to any of Claims 1 to 4 characterized in that it additionally contains from 1% to 10% of an emollient.

6. A solid stick composition according to Claim 5 characterized in that said emollient is selected from ethylene oxide and/or propylene oxide condensation products, fatty acid esters, fatty alcohol esters, water insoluble ethers, and mixtures thereof.

Best Available Copy

7. A solid stick composition according to any of Claims 1 to 6 which additionally contains from 10% to 70% of an astringent antiperspirant salt.
8. A solid stick composition according to Claim 7 characterized in that said astringent antiperspirant salt is a complex of aluminium chlorhydroxide, a zirconium compound and glycine.
9. A solid stick composition according to any of Claims 1 to 8 characterized in that said C<sub>20</sub> and/or longer chain alcohols are present at from 1% to 2% of the total long-chain higher fatty alcohol.

(19)



Europäisches Patentamt  
European Patent Office  
Office européen des brevets

(11) Publication number:

**0 117 070  
A3**

(12)

## EUROPEAN PATENT APPLICATION

(21) Application number: 84300373.2

(51) Int. Cl.<sup>4</sup>: **A 61 K 7/32**

(22) Date of filing: 23.01.84

(30) Priority: 26.01.83 US 460973

(43) Date of publication of application:  
29.08.84 Bulletin 84/35

(88) Date of deferred publication of search report: 18.12.85

(84) Designated Contracting States:  
AT BE CH DE FR GB IT LI NL

(71) Applicant: **THE PROCTER & GAMBLE COMPANY**  
301 East Sixth Street  
Cincinnati Ohio 45201(US)

(72) Inventor: **May, William George**  
4869 Beechwood Road  
Cincinnati Ohio 45244(US)

(74) Representative: **Brooks, Maxim Courtney et al,**  
Procter & Gamble (NTC) Limited Whitley Road  
Longbenton  
Newcastle-upon-Tyne NE12 9TS(GB)

(54) **Cosmetic sticks.**

(57) Solid stick compositions comprising a long-chain fatty alcohol having from about 8 to about 18 carbon atoms in the chain, volatile silicone and C<sub>20</sub> or longer chain alcohols at levels of from about 1% to about 3% of the total long-chain fatty alcohol present in the compositions. These solid sticks have improved strength and hardness without undue brittleness.

**EP 0 117 070 A3**



European Patent  
Office

# EUROPEAN SEARCH REPORT

0117070

Application number

EP 84 30 0373

DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl. 3)
X	US-A-3 641 239 (S.R. MOHRLOK) * Column 2, line 23 - column 3, line 56; column 16, lines 21-34,35-50; column 17, line 4 *	1-6,9	A 61 K 7/32 A 61 K 7/38
X,D	--- US-A-4 280 994 (M.E. TURNEY) * Column 2, line 29 - column 3, line 19; column 3, lines 29-49; claims 1,11 *	1-9	
X	--- GB-A-2 062 466 (NAARDEN INTERNATIONAL) * Page 2, lines 35-47,58-100; example 8 *	1-9	
Y	--- US-A-4 151 272 (GEARY et al.) * Whole document *	1-9	TECHNICAL FIELDS SEARCHED (Int. Cl. 3)
Y	--- US-A-2 942 008 (I.I. LUBOWE) * Column 1, line 35; column 2, line 10-25,49-50,60-63; colimn 6, lines 16-35,40-45 *	1-9	A 61 K
Y,D	--- US-A-4 126 679 (DAVY et al.) * Claims 1-16 *	1-9	
	--- -/-		
The present search report has been drawn up for all claims			
Place of search THE HAGUE		Date of completion of the search 07-08-1985	Examiner FISCHER J.P.
<b>CATEGORY OF CITED DOCUMENTS</b>			
X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document		T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons & : member of the same patent family, corresponding document	

EPO Form 1503 03.82



European Patent  
Office

# EUROPEAN SEARCH REPORT

0117070

Application number

EP 84 30 0373

Page 2

DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl. 3)
Y	FR-A-1 190 002 (WICKHEN PRODUCTS) * Page 1, left-hand column, lines 24-38; page 2, right-hand column, lines 40-57; page 3 left-hand column, lines 2-26 *	1-9	
Y,D	--- US-A-4 229 432 (GERIA) * Whole document *	1-9	
Y	--- DE-A-2 352 266 (HENCKEL) * Page 8, examples 4,5 * -----	1-9	
The present search report has been drawn up for all claims			TECHNICAL FIELDS SEARCHED (Int. Cl. 3)
Place of search THE HAGUE		Date of completion of the search 07-08-1985	Examiner FISCHER J.P.
<div>CATEGORY OF CITED DOCUMENTS</div> <div><div>X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document</div><div>T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons &amp; : member of the same patent family, corresponding document</div></div>			

EPO Form 1803, 03.82

**THIS PAGE BLANK (USPTO)**



**This Page is Inserted by IFW Indexing and Scanning  
Operations and is not part of the Official Record**

**BEST AVAILABLE IMAGES**

Defective images within this document are accurate representations of the original documents submitted by the applicant.

Defects in the images include but are not limited to the items checked:

- ☐ **BLACK BORDERS**
- ☐ **IMAGE CUT OFF AT TOP, BOTTOM OR SIDES**
- ☐ **FADED TEXT OR DRAWING**
- ☐ **BLURRED OR ILLEGIBLE TEXT OR DRAWING**
- ☐ **SKEWED/SLANTED IMAGES**
- ☐ **COLOR OR BLACK AND WHITE PHOTOGRAPHS**
- ☐ **GRAY SCALE DOCUMENTS**
- ☐ **LINES OR MARKS ON ORIGINAL DOCUMENT**
- ☐ **REFERENCE(S) OR EXHIBIT(S) SUBMITTED ARE POOR QUALITY**
- ☐ **OTHER:** \_\_\_\_\_

**IMAGES ARE BEST AVAILABLE COPY.**

**As rescanning these documents will not correct the image problems checked, please do not report these problems to the IFW Image Problem Mailbox.**

THIS PAGE BLANK (USPTO)